8 GAS CHROMATOGRAPH-MASS SPECTROMETER (GC-MS)

A. GENERAL

a. The GC-MS column shall be an HP-1MS, 30 m X 0.25 mm ID X 0.25 μm film thickness or equivalent.

b. The primary operator, or their designee, shall tune the instrument using perfluorotributylamine (PFTBA) with the manufacturer's maximum sensitivity autotune or ATUNE each day unless there is no anticipated usage of the instrument for that day.

   i. The autotune report must be examined by a qualified analyst to ensure that all parameters are within an acceptable range.

   ii. All hard copies of autotune reports will be maintained in the instrument's quality control file for one year.

   iii. The peak width at half height (pW50) will be changed from the factory setting of 0.6 to a value of 0.5.

c. The primary operator, or their designee, shall collect and print a background spectrum each day unless there is no anticipated usage of the instrument for that day.

   i. All hard copies of the daily background will be maintained in the instrument's quality control file for one year.

d. The septum will be changed at least once per month, or as needed.

e. The pressure of the compressed gas cylinder providing research grade helium to the system shall be checked on a regular basis, and the tank will be changed when the pressure is less than 300 psi.

f. When noticeable changes occur in the instrument's performance as noted in the autotune or standard samples that have been run (e.g., air leaks, decreased resolution, decreased sensitivity), the following items shall be checked as necessary: septum, column nut, GC-MS interface nut, injection port liner, column, autosampler and MS source. Any corrective action or maintenance will be recorded in the log book maintained for the instrument.

g. Archive data files on a monthly basis.

h. Preventative maintenance will be performed by qualified personnel per service contract for the instrument.

B. SAMPLE ANALYSIS

a. Samples shall be characterized as much as possible prior to injection into the instrument. Injection volumes (concentration range), chromatographic conditions, and data acquisition parameters must be determined prior to injection.

b. For both manual and autosampler injections, a blank, standard and sample must be run under the same chromatographic conditions and data acquisition parameters, as appropriate.

c. When running multiple samples, either manually or by autosampler, an instrument blank shall be run between injections.

d. For headspace samples, use disposable syringes.

i. The blank shall be appropriate for the sample being run. For example, if the sample is in a headspace vial, inject a headspace vial blank.

e. For liquid injections, the blank shall be the solvent used for the case samples and is run as appropriate.

f. Retention times for single component analytes are expected to agree with the standard within 2 seconds (± 2 sec.) or 0.03 minutes.

g. A background subtracted mass spectrum and tabulation should be collected. Reference to in-house and/or published standard spectra should be done to verify sample identification.

h. Generally speaking, appropriate volumes for diluted standards and case samples range from 0.3 to 3.0 µL, for headspace samples, up to 3.0 mL.

i. Generally speaking, a 50:1 split is used for diluted standards and "strong" case samples, a 20:1 split is used for weaker case samples, and a splitless injection may be used for even weaker samples.

i. Routinely, a starting place for injections would be: 1.0 µL at 50:1 for diluted standards and "strong" case samples, and 2.0 µL at 20:1 for extracted case samples.

j. The selected ion monitoring (SIM) mode may only be used after exhausting the use of the full scan (TIC) mode in conjunction with sample concentration and maximum injection volume. Before SIM-ing, determine that there is an indication that the compound(s) of interest may be present at the appropriate retention time(s) with the appropriate major ions.

C. DOCUMENTATION - SINGLE PEAK IDENTIFICATION

a. At a minimum, include the following:

i. A notation that the blanks before/between injections are acceptable (no peaks in the region of interest)
ii. The one page TIC and spectrum, and the normalized tabulation for the sample; the library printout may be included
iii. The one page TIC and spectrum, and the normalized tabulation for the standard
iv. The solvent blank TIC showing no peaks in the region of interest at a scale of very low abundance OR a printout of a time block around the retention time of interest showing that the extracted ions of the compound of interest are not present, as appropriate.
v. If there is an air peak present the peak may be verified on screen and a notation hand-written on the TIC of the sample OR you may print the TIC and spectrum of the air peak – do not print the tabulation and library search.
vi. If the sample is weak, it will be necessary to demonstrate that the blank before the sample is acceptable. Include the blank demonstrating that the ions of interest are not present.
vii. GC-MS Conditions Sheet (Appendix 19)

♦End

ATTACHMENT 1

AUTO1-50; AUTO2-20; AUTO3-20; AUTO2-00; MANL-50; MANL-20; MANL-00 METHODS:

Inlet Temperature:	290°C
Initial Oven Temperature:	60°C
Hold time @ Initial Temperature:	3 min
Temperature Ramp #1:	8 °C/min
Temperature #2:	140 °C
Hold time @ Temperature #2:	-0-
Temperature ramp #2:	20°C/min
Final Oven Temperature:	300°C
Final Hold time:	4 min
Transfer Line Temperature:	300°C

Scan conditions: m/z range: 0 to 15 minutes: 14 – 400 amu, Sampling = 3; @15 min: 14-600, Sampling = 2
Solvent delay: ≈ 1.8 minutes
Column flow: 0.6 ml/min

AUTO1-50 has a split flow of 50 mL/min, uses the autosampler and injects 1µL
AUTO2-20 has a split flow of 20 mL/min, uses the autosampler and injects 2µL
AUTO3-20 has a split flow of 20 mL/min, uses the autosampler and injects 3µL
AUTO2-00 is a splitless injection, uses the autosampler and injects 2µL
MANL-50 has a split flow of 50 ml/min and requires a manual injection
MANL-20 has a split flow of 20 ml/min and requires a manual injection
MANL-00 is a splitless injection and requires a manual injection

HEADSPACE METHOD

Manual Injection, 20:1 split:

Inlet Temperature:	290 °C
Initial Oven Temperature:	35 °C
Hold Time @ Initial Temperature:	4 min
Temperature Ramp:	10 °C/min
Final Oven Temperature:	100°C
Hold time @ Final Temperature:	0 min
Transfer Line:	300°C

Scan conditions: 14 – 150 amu; Sampling = 3
Solvent delay: none
Column flow: 0.6 ml/min

HEAVY OIL METHOD

Manual injection, 20:1 split:

Inlet Temperature:	290 °C
Initial Oven Temperature:	150 °C
Hold Time @ Initial Temperature:	0 min
Temperature Ramp:	15 °C/min
Final Oven Temperature:	325°C

Hold time @ Final Temperature: 4 min
Transfer Line: 325°C

Scan conditions: 14 – 600 amu; Sampling = 3
Solvent delay: ≈ 1.8 minutes
Column flow: 0.6 ml/min

♦End